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Indian Standard

SPECIFICATION FOR
p-NITROTOLUENE *o*-SULPHONIC ACID

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 1

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May 1968

Indian Standard
SPECIFICATION FOR
p-NITROTOLUENE *o*-SULPHONIC ACID

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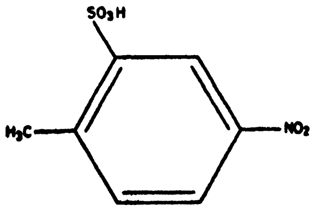
Indian Standard
SPECIFICATION FOR
p-NITROTOLUENE *o*-SULPHONIC ACID

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 28 October 1967, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Chemical Division Council.

0.2 *p*-Nitrotoluene *o*-sulphonic acid is an intermediate used in the synthesis of dyes as well as for the manufacture of diaminostilbene disulphonic acid. It has the following structural formula:



p-Nitrotoluene *o*-Sulphonic acid
(Molecular weight 217·2)

0.3 This standard is one of a series of Indian Standards on dye intermediates. Other standards in the series are given on page 11.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final values, observed or calculated, expressing the results of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off values should be the same as that of the specified values in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for *p*-nitrotoluene *o*-sulphonic acid.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of a yellow to pale brown crystalline powder or paste.

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*Rules for rounding off numerical values (*revised*).

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR *p*-NITROTOLUENE *o*-SULPHONIC ACID

Sl No.	Characteristic	Requirement	Method of Test Ref to Cl No. in Appendix A
(1)	(2)	(3)	(4)
i)	Assay on dried sample (calculated on molecular weight 217·2), percent by weight, *Min*	95·0	A-1
ii)	Matter insoluble in sodium carbonate solution, percent by weight, *Max*	0·1	A-2
iii)	Free sulphuric acid, percent by weight, *Max*	5·0	A-3

3. PACKING AND MARKING

3.1 Packing — Unless otherwise agreed, the material shall be packed in suitable drums (*see* IS : 2552-1963*) lined with a suitable polyethylene film.

3.2 Marking — The container shall be securely closed and marked with the following information:

a) Name of the material;
b) Name of the manufacturer;
c) Lot or batch number;
d) Gross, net and tare weight; and
e) Registered trade-mark, if any.

3.2.1 Each container shall also be marked with the minimum cautionary notice 'TOXIC; HANDLE WITH CARE'.

3.2.2 The containers may also be marked with the ISI Certification Mark.

Note — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act, and the Rules and Regulations made thereunder. Presence of this mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard, under a well-defined system of inspection, testing and quality control during production. This system, which is devised and supervised by ISI and operated by the producer, has the further safeguard that the products as actually marketed are continuously checked by ISI for conformity to the standard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

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*Specification for steel drums (galvanized and ungalvanized).

4. SAMPLING

4.1 The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in Appendix B.

5. TEST METHODS

5.1 Tests shall be conducted according to the methods prescribed in Appendix A. References to relevant clauses are given in col 4 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise pure chemicals and distilled water (*see* IS : 1070-1960*) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

(*Table* 1, and *clause* 5.1)

METHODS OF TEST FOR *p*-NITROTOLUENE *o*-SULPHONIC ACID

A-1. ASSAY

A-1.0 Outline of the Method — The nitro group is reduced with zinc and concentrated hydrochloric acid. The resulting amino group is estimated by diazotisation with standard sodium nitrite solution.

A-1.1 Reagents

A-1.1.1 *Zinc Dust*

A-1.1.2 *Standard Sodium Nitrite Solution* — 0·2 N freshly standardized.

A-1.1.3 *Concentrated Hydrochloric Acid* — *see* IS : 265-1962†.

A-1.1.4 *Potassium Bromide* — analytical reagent grade.

A-1.1.5 *Starch-iodide Test Papers*

A-1.2 Procedure — If the original material is in the form of a paste, dry the sample by heating it to 110°C till a constant weight is obtained. Dissolve, about 20 g of the material, accurately weighed, in hot water. Cool to room temperature and dilute up to mark in a 1-litre measuring flask. From this solution pipette 200 ml into a wide-mouthed conical flask and

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*Specification for water, distilled quality (*revised*).

†Specification for hydrochloric acid (*revised*).

add 20 g zinc dust in the flask. Close the flask with a rubber stopper through which a reflux condenser and a long stem separating funnel are inserted. Allow it to stand at room temperature for 10 to 15 minutes with occasional shaking. Add dropwise with thorough shaking 100 ml of concentrated hydrochloric acid from the separating funnel. When the reaction subsides, place the flask on a steam bath and allow it to stand on the bath for 30 minutes with occasional shaking. Then wash the inner sides of condenser and cool the solution in ice water. Filter the solution through cotton wool and wash the residue thoroughly with water till the washing shows no acidity to Congo red paper.

Transfer the filtrate and washings to a 1·5-litre beaker and cool to about 10°C by adding ice directly. Then add 5 g of potassium bromide and stir mechanically. Titrate with standard sodium nitrite solution until an immediate blue ring test on starch-iodide test paper is produced, which may be obtained repeatedly during a period of 10 minutes without further addition of nitrite solution.

A-1.3 Calculation

$$\text{Assay, percent by weight (Calculated on molecular weight 217·2)} = \frac{108·6 \times V \times N}{W}$$

where

V = volume in ml of standard sodium nitrite solution consumed,

N = normality of the standard sodium nitrite solution, and

W = weight in g of the material taken for the test.

A-2. DETERMINATION OF MATTER INSOLUBLE IN SODIUM CARBONATE SOLUTION

A-2.1 Reagent

A-2.1.1 *Sodium Carbonate Solution* — 10 percent (*w/v*), approximately.

A-2.2 Procedure — Weigh accurately about 10 g of the sample. Transfer this to a 500 ml beaker with 100 ml water. Dissolve it in just the required volume of sodium carbonate solution. Filter through a tared sintered glass crucible, wash with water, dry in an oven at about 100°C to constant weight. Cool in a desiccator and weigh.

A-2.3 Calculation

$$\text{Matter insoluble in sodium carbonate solution, percent by weight} = \frac{W_1}{W_2} \times 100$$

where

W_1 = weight in g of the residue, and
W_2 = weight in g of the material taken for the test.

A-3. DETERMINATION OF FREE SULPHURIC ACID

A-3.0 Outline of the Method — The free sulphuric acid is precipitated as barium sulphate which is washed, dried and weighed, and from this the percent by weight of free sulphuric acid present is determined.

A-3.1 Reagents

A-3.1.1 *Barium Chloride Solution* — 10 percent (*w/v*), approximately.

A-3.1.2 *Concentrated Hydrochloric Acid* — *see* IS : 265-1962*.

A-3.2 Procedure — Accurately weigh about 10 g of the sample. Dissolve in water and make up to 250 ml in a volumetric flask. If necessary, filter this solution into a clean dry beaker through a dry filter paper and use the filtrate for the determination of acidity. Pipette 50 ml aliquot of the sample into a 600 ml beaker and add 200 ml of water. Then add 2 ml of concentrated hydrochloric acid and heat to boiling. Remove the solution from the source of heat and add 25 ml of barium chloride solution in a fine stream with constant stirring. Add the reagent in slight excess of that required for precipitation [10 ml will precipitate about 0·4 g of sulphates (as SO_4)]. Place the beaker in a steam-bath for 4 hours and allow the precipitate to settle. If the supernatant liquid is still turbid after 2 hours, allow the solution to stand overnight. Check completeness of precipitation by adding one or two drops of barium chloride solution to the clear supernatant liquor. If more precipitate is formed, continue the addition of hot barium chloride solution until an excess has been added.

Wash the precipitate by decantation with hot water till washings are free from chlorides. Filter the precipitate through a weighed sintered glass crucible (G No. 4) or a Gooch crucible or a filter paper (Whatman No. 42 or equivalent). If the filtration has been done through a sintered glass crucible, dry the precipitate at 110° ± 5°C. In the case of Gooch crucible, dry the precipitate and ignite it over a burner or in the muffle furnace at 600° to 700°C for half an hour. In the case of filter paper, after drying, the filter paper containing the precipitate is burnt out on a low flame and then ignited over a burner or in a muffle furnace at 600° to 700°C for half an hour. Cool in a desiccator and weigh to constant weight.

---

*Specification for hydrochloric acid (*revised*).

A-3.3 Calculation

$$\text{Free sulphuric acid, percent by weight (on molecular weight 98)} = \frac{w \times 210{\cdot}08}{W}$$

where

w = weight in g of barium sulphate, and
W = weight in g of the material taken for the test.

APPENDIX B

(*Clause* 4.1)

SAMPLING OF *p*-NITROTOLUENE *o*-SULPHONIC ACID

B-1. GENERAL REQUIREMENTS OF THE SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall be taken at a place protected from damp air, dust and soot.

B-1.2 Sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples, from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling, shall be mixed as thoroughly as possible, by suitable means.

B-1.5 The samples shall be placed in a clean, dry and air-tight glass or other suitable containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, year of manufacture, and other important particulars of the consignment.

B-1.8 Samples shall be stored in a cool and dry place.

B-2. SAMPLING INSTRUMENT

B-2.1 The sampling instrument is a closed type sampling tube, undivided (Fig. 1) consisting of two concentric cylindrical tubes made of a metal which is not affected by the material being sampled (preferably of stainless steel) closely fitting into each other throughout their entire length so that one tube may be rotated within the other, a suitable handle being provided for the purpose. Longitudinal openings of about one third the circumference are cut in both tubes throughout their length. In one position the two openings coincide and admit the material into the hollow inner tube. By rotating the inner tube through 180° the opening is tightly closed and a " core " of material being enclosed therein may be withdrawn. This type of sampler is usually provided with locking arrangement so that the tubes are held together in any desired position. The outer tube is provided with a sharp conical end to facilitate penetration but the base of the cone shall be closed so that no material is entrapped in this portion. The height of the cone shall be equal to its base diameter. The whole instrument shall be of sufficient length to penetrate an entire diagonal of the container being sampled. The diameter of the inner cylindrical space may vary from 20 to 40 mm proportionately to the length. A length of 150 cm and a diameter of 30 mm caters for most needs.

B-2.1.1 *Use of Sampling Instrument* — The instrument is inserted in closed position in an oblique direction till it touches bottom. The material is admitted by rotating and opening the tubes and finally closing them, withdrawing the sample in this process. In case the minimum quantity of material required for test from each container is more than the capacity of the instrument, further ' cores ' shall be taken from different parts of the same container such that they are at least 75 mm in the case of kegs, drums, bags, etc, and 25 mm in the case of small containers, from the wall of the container. In all cases the instrument shall be inserted till it touches bottom so that an entire cross-section is withdrawn.

B-3. SCALE OF SAMPLING

B-3.1 Lot — In any consignment all the containers of the same size and drawn from the same batch of manufacture, shall constitute a lot. If a consignment is known to consist of different batches of manufacture or of different sizes of containers, the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

B-3.2 Tests shall be conducted on each lot separately for ascertaining its conformity to the requirements of this specification. The number of containers to be chosen at random from the lot for this purpose shall depend on the size of the lot and shall be in accordance with col 1 and 2 of Table 2.

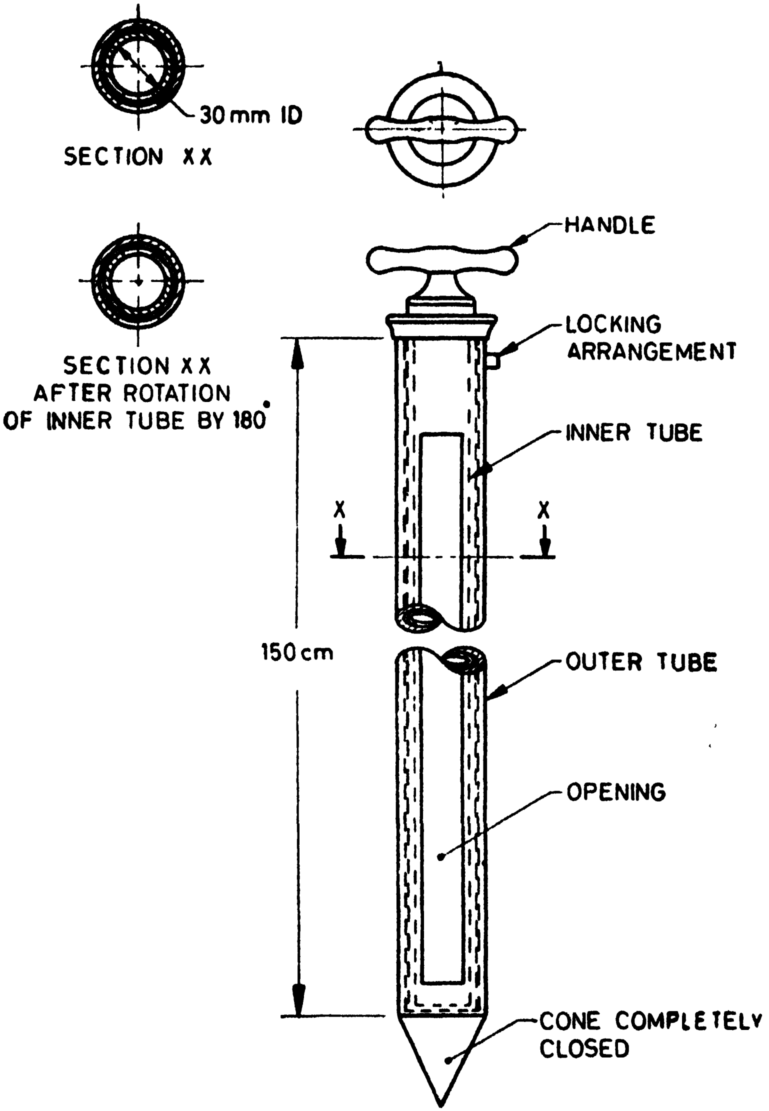

Fig. 1 Closed Type Sampling Tube, Undivided

TABLE 2 SCALE OF SAMPLING

Lot Size N (1)	No. of Containers to be Selected n (2)
Up to 100	5
101 „ 200	6
201 „ 300	7
301 „ 400	8
401 „ 500	9
501 and above	10

Note — In case of very small lots all the containers shall be selected.

B-3.3 The containers shall be chosen at random from the lot with the help of a suitable random number table. In case no such table is available, the following procedure is recommended for use.

> Starting from any container from the lot, count them as 1, 2, 3,... up to r and so on in one order where r is the integral part of N/n (N being the number of containers in the lot and n being the number of containers to be selected). Every rth container thus counted shall be withdrawn to give samples for tests.

B-4. PREPARATION OF TEST SAMPLES

B-4.1 From each of the containers selected in **B-3.2**, a small representative portion of the material from different parts of the container shall be drawn with the help of a suitable sampling instrument (*see* **B-2**).

B-4.2 Out of these portions a small but equal quantity of material shall be taken and mixed thoroughly to form a composite sample. The composite test sample shall be divided into three equal parts one for the purchaser, another for the supplier and the third to be used as a referee sample.

B-4.3 The remaining portion of the material from each of the containers (*see* **B-4.2**) shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the containers sampled shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

B-4.4 All the individual and composite samples shall be transferred to separate containers and shall be sealed and marked with full identification particulars given under **B-1.7**.

B-4.5 The referee test samples consisting of a composite sample and a set of *n* individual samples, shall bear the seals of both the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier to be used in case of any dispute between the two.

B-5. NUMBER OF TESTS

B-5.1 Assay shall be conducted on each of the individual samples.

B-5.2 Tests for the determination of all other characteristics given under Table 1 shall be conducted on the composite samples.

B-6. CRITERIA FOR CONFORMITY

B-6.1 For Individual Samples — The lot shall be declared as conforming to the requirements of assay, if the test results satisfy the relevant requirements given under Table 1.

B-6.2 For Composite Samples — For declaring the conformity of a lot to the requirements of all other characteristics tested on the composite sample (*see* **B-4.2**), the test results for each of the characteristics shall satisfy the relevant requirements given in Table 1.

INDIAN STANDARDS

ON

Dye Intermediates

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2741-1964	β-Naphthol	3·00
2744-1964	α-Naphthylamine	3·00
2833-1964	Aniline, technical	3·00
3229-1965	Naphthionic acid (sodium salt)	2·50
3242-1965	β-Oxy naphthoic acid (BON ACID)	3·00
3562-1965	*p*-Nitrotoluene, technical	4·50
4265-1967	4-4′ Diaminostilbene 2-2′ disulphonic acid	4·00
4334-1967	*o*-Chloroaniline	5·00
4335-1967	*m*-Chloroaniline	5·00
4336-1967	*p*-Chloroaniline	5·50

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TO

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Alteration

(First cover, pages 1 and 2, title) - Substitute the following for the existing title:

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SPECIFICATION FOR 4-NITROTOLUENE-3-SULPHONIC ACID'

(PCDC 11)

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